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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification⁶ : A23L 1/22, 2/68, A23P 1/02</p>	<p>A1</p>	<p>(11) International Publication Number: WO 98/53705 (43) International Publication Date: 3 December 1998 (03.12.98)</p>
<p>(21) International Application Number: PCT/GB98/01498 (22) International Filing Date: 22 May 1998 (22.05.98) (30) Priority Data: 97/4675 28 May 1997 (28.05.97) ZA (71) Applicant (for all designated States except MW US): SENTRA- CHEM LIMITED [ZA/ZA]; 5 Protea Place, Off Fredman Drive, Sandown, Sandton 2196 (ZA). (71) Applicant (for MW only): BARLOW, Roy, James [GB/GB]; J.A. Kemp & Co., 14 South Square, Gray's Inn, London WC1R 5LX (GB). (72) Inventor; and (75) Inventor/Applicant (for US only): FOWLDS, Robert, Walter, Rycroft [ZA/ZA]; 7 Ocean Villa, 47 Gusbrown Road, Warner Beach 4126 (ZA). (74) Agents: MARSHALL, Monica, Anne et al.; J.A. Kemp & Co., 14 South Square, Gray's Inn, London WC1R 5LX (GB).</p>		<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: THE PRODUCTION OF A FOOD ACID MIXTURE CONTAINING FUMARIC ACID</p> <p>(57) Abstract</p> <p>A method for the production of a food acid includes the steps of combining fumaric acid with an organic acidic material in an aqueous medium to produce a mixture and drying the mixture to produce a food acid comprising a particulate material containing fumaric acid and the organic acidic material, the quantity of fumaric acid and the quantity of the organic acidic material being selected so that the fumaric acid makes up between about 5 % and 95 % of the particulate material.</p>		

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THE PRODUCTION OF A FOOD ACID MIXTURE CONTAINING FUMARIC ACID

THIS INVENTION relates to a method for the production of a food acid and to a food acid produced in accordance with the method.

According to a first aspect of the invention there is provided a method for the production of a food acid, the method including the steps of

5 combining fumaric acid with an organic acidic material in an aqueous medium to produce a mixture; and

drying the mixture to produce a food acid comprising a particulate material containing fumaric acid and the organic acidic material, the quantity of fumaric acid and the quantity of the organic acidic material being selected so that the fumaric
10 acid makes up between about 5% and 95% of the particulate material.

The percentage compositions given in this specification refer to percentage compositions by mass.

Preferably, the quantity of fumaric acid and the quantity of the organic acidic material will be selected so that the fumaric acid makes up between about
15 40 and 60% of the particulate material, and more preferably between about 45 and 55%.

The fumaric acid may be cold water soluble (or CWS) fumaric acid. Cold water soluble fumaric acid refers to finely ground fumaric acid which includes a small amount of a wetting agent of the type marketed as CWS fumaric acid by
20 Haarmann and Reimer or a granular fumaric acid material of the type marketed by NCP Food Products as granular cold water soluble fumaric acid.

The organic acidic material may be selected from malic acid, tartaric acid, citric acid, lactic acid, ascorbic acid and mixtures of any two or more thereof.

Preferably the organic acidic material will be a mixture of malic acid and tartaric acid. The organic acids are thus water-soluble edible acids i.e. acids permitted in foods.

The quantity of malic acid may be selected so that the malic acid
5 makes up about 40 - 60% of the particulate material and preferably about 50%.
The quantity of tartaric acid may be selected so that the tartaric acid makes up about 3 - 10% of the particulate material and preferably about 5%.

The method may thus involve admixing finely divided fumaric acid with an aqueous solution of malic acid and tartaric acid. The admixing step may
10 include grinding the fumaric acid in the presence of the aqueous solution of the malic acid and tartaric acid to provide the slurry or grinding the fumaric acid in the aqueous medium and then adding or blending the malic acid and tartaric acid into the milled fumaric acid slurry or spraying an aqueous solution of malic acid and tartaric acid, or spraying separate solutions of malic acid and tartaric acid, onto
15 fumaric acid in powder form.

Thus the combining step may include admixing finely divided fumaric acid with an aqueous solution of malic acid and tartaric acid. Instead, the combining step may include grinding the fumaric acid in the presence of an aqueous solution of malic acid and tartaric acid to produce a slurry. Instead, the
20 combining step may include grinding the fumaric acid in an aqueous medium to produce a milled fumaric acid slurry and then adding or blending the malic acid and tartaric acid into the milled fumaric acid slurry. Instead, the combining step may include spraying an aqueous solution of malic acid and tartaric acid onto fumaric acid in powdered form. Instead, the combining step may include spraying separate
25 aqueous solutions of malic acid and tartaric acid onto fumaric acid in powder form.

The grinding step may be conducted in a wet mill so that the mixture is in the form of a slurry in which the particle size of essentially all the solid material in the slurry is not greater than 150 microns (100 mesh) and the average

particle size is between about 100 and 25 microns (150 - 500 mesh). Preferably, the grinding step is conducted so that the particle size of essentially all of the solid material in the slurry is not greater than 100 microns (150 mesh) and the average particle size is between about 75 and 25 microns (200 - 500 mesh).

- 5 The drying step may include spray drying or spray granulating the slurry. Thus the drying step may be selected from spray drying and spray granulating. In particular, the drying step may be a spray granulating step which is conducted in a fluid bed granulator by a continuous or a batch process to give a free-flowing non-dusting product. The spray granulation step may be controlled
- 10 such that granules of 20 - 60 mesh, preferably 30 - 45 mesh are produced directly, or are selected from the product by screening, with the rejected material being reprocessed.

 The final particle size is typically controlled by screening or sifting, to remove oversize particles which are then ground finely in a mill and returned to the

15 fluidized bed of the granulator. Undersized particles are also removed by screening or sifting and are then returned to the fluidized bed. Further, when the drying step is conducted by a continuous process, a certain amount of material segregation of the particles takes place as the smaller particles are lifted higher and tend to come into contact with more of the fresh solution/slurry being sprayed into the bed, and

20 consequently grow in preference to the larger particles which remain in the lower reaches of the bed. The overflow of the bed is generally situated near the bottom of the bed to remove the larger particles. In this way, the final particle size is controlled between about 20 and 100 mesh (840 -150 microns) and ideally between about 24 and 60 mesh (700 - 250 microns).

- 25 The particulate material may, after the spray granulation step, be dried to a moisture level of less than 0,5% and preferably less than 0,25%.

 Thus, the mixture may be dried to produce a particulate material having a moisture level of less than 0,5%, and preferably less than 0,25%.

The combining step may be conducted in the presence of a wetting agent or surfactant. The wetting agent may, for example, be dissolved in the aqueous medium so that the particulate material which is produced is uniformly mixed with the wetting agent. The admixing step may, further, be conducted in
5 the presence of an anti-foaming agent. The anti-foaming agent may also be dissolved in the aqueous solution.

The wetting agent may be a liquid wetting agent. Such a liquid wetting agent will typically be edible and essentially tasteless. It may, for example, be a liquid alkyl sulphosuccinate such as dioctyl sodium sulphosuccinate,
10 sodium lauryl sulphate, Tween (trade name) or any other suitable edible wetting agent or surfactant. The anti-foaming agent may be a silicone anti-foaming agent. It may, for example, be a food grade silicone oil anti-foaming agent.

The quantity of the wetting agent may be selected so that it comprises about 0,1 - 0,5% of the final product i.e. of the food acid. The quantity
15 of the anti-foaming agent may be selected so that it controls foam during the admixing and drying steps and comprises about 2 - 20 ppm preferably about 5 - 15 ppm and most preferably about 10 ppm of the final product i.e. of the food acid.

The method may include the further step of incorporating an additive
20 selected from flavourants, colourants, sweeteners and mixtures of two or more thereof in the food acid. Typically the additive will be sprayed onto the mixture during the spray-drying or spray-granulating step.

The invention extends to a food acid in particulate form produced by a method as hereinbefore described.

25 The invention extends further to a food acid in particulate form, the food acid comprising a composite material which includes fumaric acid in an

amount of about 5% - 95%, malic acid in an amount of about 5 - 95% and tartaric acid in an amount of about 3 - 15%.

The invention is now described, by way of Example, with reference to the accompanying Examples and the Figures in which the words "composite acid" and "composite" refer to the food acid of the invention. In the figures

Figure 1 shows the expected tartness profile of the composite acid;

Figure 2 shows the tartness profile of citric acid;

Figure 3 shows the tartness profile of malic acid;

Figure 4 shows the tartness profile of fumaric acid;

Figure 5 shows the tartness profile of tartaric acid;

Figure 6 shows a superposition of the tartness profiles of Figures 1 to 5 and the expected tartness profile of the composite acid;

Figure 7 shows adsorption isotherms of the composite and citric acid; and

Figure 8 shows the pH profiles of the composite and citric acid.

15

EXAMPLE 1

Malic acid (40 kg), dl-tartaric acid (4 kg) and dioctyl sulphosuccinate wetting agent (Zenith DSS, 100g) were dissolved in warm water (130kg) to produce a solution. Crystalline fumaric acid (36 kg) was added to the solution and the resultant slurry was milled in a Chicago Boiler attritor mill for two hours.

In other embodiments, the mill was an EHP Series Supermill supplied by Premier Mill or Oliver and Battle. A silicone anti-foaming agent (AF 1510, sourced from Bob Larson Silicones, five drops) was used to control foaming during the milling process. A starter-bed of crystalline tartaric acid (200g) was added to a GLATT GPCG - 60 batch spray-granulator fitted with a triple spray nozzle and the milled slurry was sprayed onto the starter bed at 70 - 130 l/h over 130 minutes so that the temperature in the fluidized bed was maintained at 50 -60°C. The mill was a 22 kW unit which was approximately 1300 mm high with a diameter of approximately 800 mm and a tip speed of the impeller of 13 m/s. The granulator settings were:

spray-air pressure 2,5 bar
inlet air temperature 100°C
air flow 2200 m³ /h

- 5 The product was then dried by further fluidization for 30 minutes and cooled by fluidization for 5 minutes in cold air to a bed temperature of 40°C.

EXAMPLE 2

- 10 Example 1 was repeated using water (60 kg) and a starter bed of the product from Example 1 (10 kg). The bed temperature during the granulation process was maintained at 52 - 57°C. The product contained 0,21% water by mass and 81,4% by mass was in the size range 841 - 250 microns. The bulk density was 690 - 774 kg/m³.

The acid taste profile of the granular product produced by the method of Example 1 is set out in Figure 1.

EXAMPLE 3

- 15 The following food grade ingredients were added to a mixing vessel fitted with an agitator at the rates indicated, and mixed:

	Tartaric Acid	50kg/hour
	Malic Acid	500kg/hour
	Fumaric Acid	450kg/hour
20	dioctyl sulphosuccinate wetting agent (70%)	2,25kg/hour
	Silicone oil anti-foaming	15g/hour
	Water	500ℓ/hour

The mixing vessel had a capacity of about 1500ℓ and was sufficiently large to allow adequate mixing and time for the soluble ingredients to dissolve. Only the fumaric acid did not dissolve, so that a slurry was formed.

5 The slurry was fed continuously into a Premier type wet mill in which it was milled. The milled slurry was then pumped directly under pressure into an APV Anhydro continuous drier/granulator unit using a positive displacement pump where granulation and initial drying of most of the moisture (down to about 1% residual) took place in a fluidized bed in the first stage of the unit. The first stage comprised a round generally squat vessel, into which the
10 slurry was pumped continuously under pressure through a nozzle into a fluid bed. This process was carried out optionally with or without atomizing compressed air. The fluid bed was suspended above a screen plate by a hot air stream. The bed was fluidised with hot air at about 65°C at superficial velocities used in fixed fluid beds, which are typically in the range of 0,3 -
15 2,4 m/s.

The height of the bed was controlled by the volume and pressure of the air entering the chamber from under the screen, while the volume and residence time of the product in the fluid bed was maintained at set conditions by allowing a certain quantity to exit the bed through a rotary outlet valve, and
20 enter a second stage. The outlet air from the first chamber was passed from the top of the vessel through a cyclone to remove entrained product fines which were returned to the fluidized bed where they came into contact with the inlet spray and other particles in the bed. This assisted in regulating the product particle size range by returning the fines to the bed
25 to grow into larger granules.

The second stage consisted of a mechanically vibrated rectangular fluid bed, or optionally a stationary fluid bed, at a slight angle, where the product is moved forward by air flows, where additional drying and subsequent cooling took place. Both the hot and cold air streams were collected with the fines
30 that were removed by the cyclone and again returned to the fluid bed of the

first chamber. Additional additives such as flavourants, colourants and/or sweeteners were optionally incorporated by spraying the additive onto the granules in the second stage.

5 The dried and cooled product from the second stage, having a low residual moisture content of typically less than 0,3% overflowed a weir which maintained a predetermined residence time, onto a screen where oversize particles were removed and ground up before being returned to the fluid bed of the first stage, together with the undersize fines which were screened out. The screening machine controlled the final particle size of the product, 10 while extraction ducts from the top of the second stage unit further classified the product by drawing off fines and dust which was removed from the air stream by the cyclone and returned to the fluid bed of the first stage granulator.

DISCUSSION

15 Food acids are generally used to provide a pleasant, refreshing acidic taste to foods, drinks and beverages. They also serve as preservation aids by lowering the pH of the foodstuff to which they are added thereby reducing the activity of possibly harmful micro-organisms. Citric acid has been used for this purpose for many years.

20 The acid taste profile of citric acid, as set out in Figure 2, can be described as a rapid "burst" of tartness which reaches a peak acid taste fairly early, with the taste falling off and disappearing quite rapidly thereafter. The sharp, strong acidic taste of citric acid tends, however, to overpower the flavour of sweeteners and flavourants in a foodstuff to which citric acid has been added.

25 The relatively rapid loss of tartness results in a bitter aftertaste if certain synthetic high-intensity sweeteners are used in the foodstuff in conjunction with the citric acid. Citric acid suffers from further disadvantages in addition to its very strong, sharp taste and short-lived tartness flavour. For example, when used in dry

powders, such as soft drink or beverage mixes, it tends to cake due to its hygroscopic nature. It is also generally not free-flowing and has an uneven particle size. Despite these drawbacks, citric acid is by far the most commonly used food acidulant.

5 Malic acid, which is as soluble as citric acid, has a tartness taste profile as shown in Figure 3. The taste profile is described as not being as sharp as citric acid but as being longer lasting. Malic acid therefore tends to mask the bitter aftertaste of synthetic sweeteners used in conjunction with malic acid. The slower increase to peak tartness does not overpower the taste of the sweeteners
10 with the result that less sweetener need be used (this has been found to be the case with aspartame). In taste tests, it has been claimed that flavourants meld more effectively with malic acid. However, the initial sharpness associated with citric acid is preferable in some end products such as citrus flavoured drinks.

 Fumaric acid has a tartness profile which is flatter than that of either
15 citric acid or malic acid but is longer lasting as can be seen in Figure 4. The longer lasting taste means that fumaric acid effectively appears to be a "stronger" food acid and consequently less fumaric acid is required to achieve a required level of tartness than is required in respect of other food acids. However, although fumaric acid is generally one of the cheapest of the food acids, it has a low solubility in
20 water and a tendency to dissolve slowly unless it is supplied for example as a very fine powder including a wetting agent i.e. so-called cold water soluble, or CWS fumaric acid of the type described above or a granular cold water soluble fumaric acid as described above. All other so-called cold water soluble fumaric acids of which the Applicant is aware are very dusty and hence unsuitable for many
25 applications.

 The tartness profile of *d,l* or *l* tartaric acid as can be seen in Figure 5 is smoother than that of citric acid. It has a peak acid taste which is higher than that of citric acid and which is longer lasting. Tartaric acid is generally the most

expensive of the commonly used food acids. The l-tartaric isomer is, however, hygroscopic and consequently tends to cake.

The tartness sensory profiles as set out in Figures 1 - 5 were gleaned from the literature. A typical method for determining these profiles is based on a software package TASTE (Ver 9.1, 1992) developed by Reading Scientific Services Limited, The Lord Zuckerman Research Centre, The University of Whiteknights, READING, UK.

In order to obtain an equi-acidic taste point of the acid composite relative to citric acid monohydrate, a panel was screened and trained. Equi-acid concentrations are the concentrations of food acids at which both have the same perceived tartness and acidity to the taste. Screening consisted of testing the panel on the four basic tastes (sweet, sour, salt and bitter) and several sets of concentration series to determine the threshold value of the group, according to Jellinek, 1985. Triangle tests were performed to determine each panel member's threshold value for each basic taste according to ASTM Standards. Subsequently the twelve members most sensitive to the bitter taste were selected as part of the acid taste panel. The sets of concentration series used in the training, were used as an indication of the range of the concentration series to be used in the equi-acidic taste analysis.

CSIR supplied the sets of concentration series for the equi-acid taste analysis. If the series was too low or too high to cover the range in which the citric acid's acidic taste point falls, a higher or lower series was requested and used. The most applicable series with a range of five concentration levels was repeated at least three times. The concentration of the citric acid was 0.2%. Samples were presented in random order pairs with the 0.2% citric acid, in ascending order of concentration, with two pairs per session with a break of 20 minutes between sessions to combat sensory panel fatigue. Instant custard was used as palate cleanser between sessions. All samples, water and custard were

served at ambient temperature (19.4 - 22.3°C). Samples were tasted in individual temperature controlled taste booths under normal white light.

The results obtained were statistically analysed by the ARC-Agrimetrics Institute, using Genstat 5, Release 3.1(1993). The equi-acid concentrations were as follows:

ACID	EQUI-ACID CONCENTRATION DETERMINED (% m/v)
Citric acid Monohydrate	0.2
Acid Composite	0.125

The above clearly shows a saving in usage. The concentrations reflect a saving of 37.5% compared to citric acid monohydrate and a saving of 33.5% when compared to citric acid anhydrous. These savings will vary depending on the flavour characteristics of the product.

These tests were followed by a comparison of the flavour characteristics of citric acid to the acid composite. The panel was trained using test acid solutions at equi-acidic taste point, and products like acetic acid, unripe bananas, cold black tea and various cordials and soft drinks. This training introduced the panellists to possible descriptive terms typical for acids. The attributes shown in Table 1 were generated and defined by the panel during training sessions. Samples were presented in the same procedure as above. The statistical analyses were collected in a spreadsheet using Quattro Pro (V 5.0) and a statistical programme, STATGRAPHICS (V 5.0) was used to analyse the data using one-way analysis of variance (ANOVA), with acid as the main effect.

TABLE 1

ATTRIBUTES	DEFINITION
SOURNESS	Basic taste on the tongue as associated with acids
SHARPNESS	A burning, harsh taste on the tongue
5 ASTRINGENT	The feeling factor on the tongue and oral cavity described as puckering/dry and associated with tannins, strong tea, unripe bananas and grape skin
BRIGHTNESS/ REFRESHING	The refreshing, clean sensation of the acid associated with water with a few drops of fresh lemon juice. (The opposite of dull, without any sparkle/"still")
DUSTINESS	Taste associated with the powdery nasal sensation of dry dust and chalk
DISPRIN	Taste associated with medicine, e.g. Disprin or aspirin
10 MOULDY	Taste associated with closed air/water spaces such as water from a closed plastic water container stored at ambient temperature for a long period of time
BITTER	Basic taste on tongue stimulated by solutions of e.g. caffeine
THROAT IRRITATING	Burning sensation felt in the throat after swallowing of the sample
AFTER-TASTE	Taste in mouth after swallowing (panellists asked to describe/comment on taste)

From the results obtained it was found that there were no significant differences between any of the sensory-related variables for citric acid (0.2% w/v) or the acid composite at their equi-acidic point.

Therefore it can be concluded that the special combination of the three acids in the composite acid can very closely match the tartness sensory

profile of citric acid across the various attributes measured at equi-acidic concentrations.

The Applicant has found that by using malic acid, fumaric acid and tartaric acid together, it is possible to overcome the claimed disadvantages of citric acid and the individual disadvantages associated with malic, fumaric and tartaric acids and to provide a composite granule, containing each of the acids, which has an improved tartness taste profile. It is expected that the sensory tartness profile of the composite acid compared to the other food acids will closely resemble that shown in Figure 6. The invention thus allows the replacement of hygroscopic citric acid with a non-hygroscopic granular composite mixture of malic acid, dl-tartaric acid (or l-tartaric acid if dl-tartaric acid is not a permitted food acid or is not available) and fumaric acid (all of these acids are food grade and non-hygroscopic in the composite granular form of the present invention). Empirical data clearly indicate the hygroscopic nature of citric acid compared to the relatively non-hygroscopic properties of the composite acid when tested under control conditions as by the Sorption Isotherm Measurements done by the CSIR, as can be seen in Figure 7. The small quantity of tartaric acid present in the composite granule provides a sudden "burst" of tartness similar to that of citric acid while the major portion of malic acid, which is very soluble, provides a smooth acid flavour. The slow solubility of fumaric acid can be overcome by using cold water soluble (CWS) fumaric acid and the low solubility of fumaric acid is overcome by selecting the quantity of fumaric acid in the composite granule so that the selected quantity will be soluble in the amount of water to be used.

Citric acid alone has the following drawbacks. It is hygroscopic and tends to cake. It is not free-flowing, has an uneven particle size, an overpowering initial sharp taste and a short-lived tartness flavour.

Furthermore, blends of acids generally have an uneven distribution of components and separation (stratification) of the components due to differing particle size and differing specific gravity frequently takes place. Such blends also have an irregular appearance.

The presence of fumaric and malic acids in the granular product also results in a saving in the overall quantity of acidulant required when compared to citric acid and provides a longer lasting acid flavour.

TABLE 2

Mean Scores for Citric Acid in comparison to Acid Composite at Equi-Acidic Point

15	SENSORY ATTRIBUTES	CITRIC ACID MONOHYDRATE	ACID COMPOSITE
	Sharpness	4.53	4.83
	Astringent	4.03	3.69
	Brightness/Refreshing	3.53	4.80
	Dustiness	1.28	1.29
	Disprin	2.44	2.40
	Mouldy	1.67	1.06
20	Bitter	1.86	1.49
	Throat Irritating	1.08	1.06
	After Taste	1.08	1.06

Where, for all the attributes: 1 = None; 8 = Extremely

It is an advantage of the invention illustrated that it provides a granular solid in which each granule is a composite granule (as opposed to a blend) which contains malic acid, tartaric acid and fumaric acid and in which the granules have a consistent particle size. The product has a good flavour resembling a natural fruit taste with a smooth long-lasting tartness and an even distribution of the various acids within the granulate. It is a further advantage of the invention illustrated that the granular product is free-flowing, dust-free and non-hygroscopic and has improved handling, packaging and shelf-life characteristics when compared with prior art acidulants of which the Applicant is aware and, furthermore, has a smooth long-lasting tartness and a lower cost than prior art food acids of which the Applicant is aware. The pH profile of the composite acid shows a consistently lower pH value at different concentrations than that achieved by citric acid as can be seen in Figure 8 and Table 3.

15

TABLE 3pH of Composite Acid and Citric at Different Concentrations

20

SOLUTION (m/v%)	pH value			
	0.25%	0.5%	0.75%	1.0%
SAMPLE				
Citric Acid (anhydrous)	2.35	2.18	2.07	2.02
Composite Acid	2.25	2.05	1.96	1.88

CLAIMS:

1. A method for the production of a food acid, the method including the steps of
combining fumaric acid with an organic acidic material in an aqueous
5 medium to produce a mixture; and
drying the mixture to produce a food acid comprising a particulate material containing fumaric acid and the organic acidic material, the quantity of fumaric acid and the quantity of the organic acidic material being selected so that the fumaric acid makes up between 5% and 95% of the particulate material.
- 10 2. A method as claimed in Claim 1, in which the quantity of fumaric acid and the quantity of the organic acidic material is selected so that the fumaric acid makes up between 40 and 60% of the particulate material.
3. A method as claimed in Claim 2, in which the quantity of fumaric acid and the quantity of the organic acidic material is selected so that the fumaric acid
15 makes up between 45 and 55% of the particulate material.
4. A method as claimed in any of the preceding claims, in which the fumaric acid is cold water soluble (or CWS) fumaric acid.
5. A method as claimed in any one of the preceding claims, in which the organic acidic material is selected from malic acid, tartaric acid, citric acid, lactic
20 acid, ascorbic acid and mixtures of any two or more thereof.

6. A method as claimed in Claim 5, in which the organic acidic material is a mixture of malic acid and tartaric acid.
7. A method as claimed in Claim 6, in which the quantity of malic acid is selected so that the malic acid makes up 40 - 60% of the particulate material.
- 5 8. A method as claimed in Claim 7, in which the quantity of malic acid is selected so that the malic acid makes up 50% of the particulate material.
9. A method as claimed in any one of Claims 6 to 8 inclusive, in which the quantity of tartaric acid is selected so that the tartaric acid makes up 3 - 10% of the particulate material.
- 10 10. A method as claimed in Claim 9, in which the quantity of tartaric acid is selected so that the tartaric acid makes up 5% of the particulate material.
11. A method as claimed in any one of Claims 6 to 10 inclusive, in which the combining step includes admixing finely divided fumaric acid with an aqueous solution of malic acid and tartaric acid.
- 15 12. A method as claimed in any one of Claims 6 to 10 inclusive, in which the combining step includes grinding the fumaric acid in the presence of an aqueous solution of the malic acid and tartaric acid to produce a slurry.

13. A method as claimed in any one of the Claims 6 to 10 inclusive, in which the combining step includes grinding the fumaric acid in an aqueous medium to produce a milled fumaric acid slurry and then adding the malic acid and tartaric acid to the milled fumaric acid slurry.
- 5 14. A method as claimed in any one of the Claims 6 to 10 inclusive, in which the combining step includes spraying an aqueous solution of malic acid and tartaric acid onto fumaric acid in powdered form.
15. A method as claimed in any one of Claim 6 to 10 inclusive, in which the combining step includes spraying separate aqueous solutions of malic acid and
10 tartaric acid onto fumaric acid in powder form.
16. A method as claimed in Claim 12 or Claim 13 in which the grinding step is conducted in a wet mill so that the particle size of essentially all of the solid material in the slurry is not greater than 150 microns (100 mesh) and the average particle size is between 100 and 25 microns (150 - 500 mesh).
- 15 17. A method as claimed in Claim 16 in which the grinding step is conducted in a wet mill so that the particle size of essentially all of the solid material in the slurry is not greater than 100 microns (150 mesh) and the average particle size is between 75 and 25 microns (200 - 500 mesh).

18. A method as claimed in any one of the preceding claims in which the drying step is selected from spray drying and spray granulating.

19. A method as claimed in Claim 18 in which the drying step is a spray granulating step which is conducted in a fluid bed granulator by a process selected
5 from continuous and batch processes to give a free-flowing non-dusting product.

20. A method as claimed in Claim 18 or Claim 19, in which the spray granulation step is controlled such that granules of 20 - 60 mesh are produced directly.

21. A method as claimed in any one of the preceding claims, in which the
10 mixture is dried to produce a particulate material having a moisture level of less than 0,5%.

22. A method as claimed in any one of the preceding claims, in which the mixture is dried to produce a particulate material having a moisture level of less than 0,25%.

15 23. A method as claimed in any one of the preceding claims in which the combining step is conducted in the presence of a wetting agent or surfactant.

24. A method as claimed in Claim 23, in which the wetting agent is dissolved in the aqueous medium so that the particulate material which is produced is uniformly mixed with the wetting agent.

25. A method as claimed in Claim 23 or Claim 24, in which the wetting
5 agent is a liquid wetting agent.

26. A method as claimed in Claim 25, in which the wetting agent is selected from dioctyl sodium sulposuccinate, sodium lauryl sulphate and Tween (trade name).

27. A method as claimed in any one of the preceding claims, which
10 includes the step of incorporating an additive selected from flavourants, colourants, sweeteners and mixtures of any two or more thereof in the food acid.

28. A food acid in particulate form produced by a method as claimed in any one of the preceding claims.

29. A food acid in particulate form, the food acid comprising a composite
15 material which includes fumaric acid in an amount of 5% - 95%, malic acid in an amount of 5 - 95% and tartaric acid in an amount of 3 - 15%.

30. A method as claimed in Claim 1, substantially as herein described and illustrated.

31. A new method for the production of a food acid substantially as herein described.
32. A food acid as claimed in Claim 28 or Claim 29 substantially as herein described and illustrated.
- 5 33. A new food acid substantially as herein described.

1/4

Fig.1.

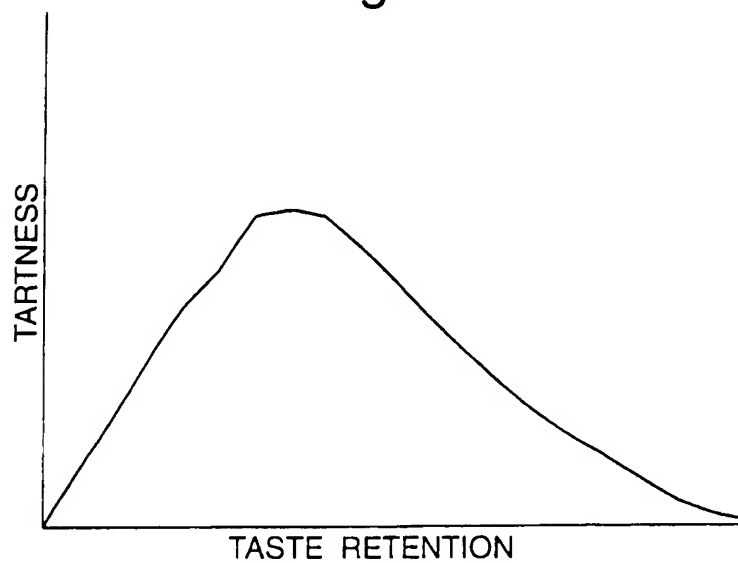


Fig.2.

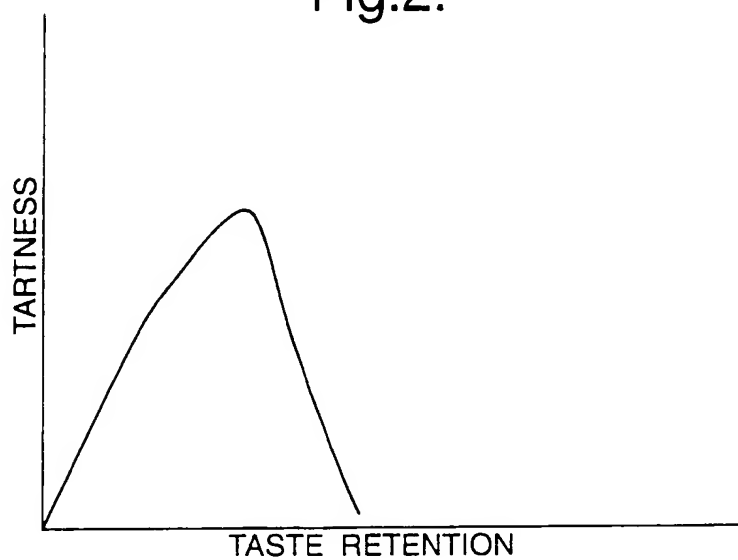


Fig.3.

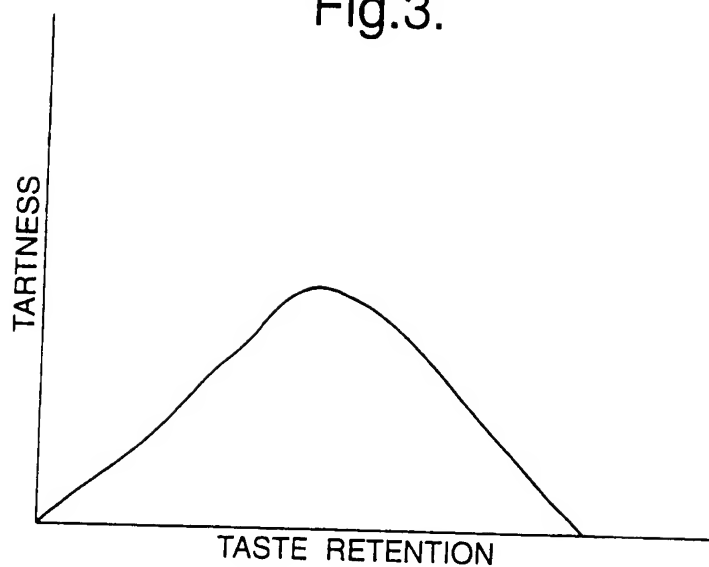
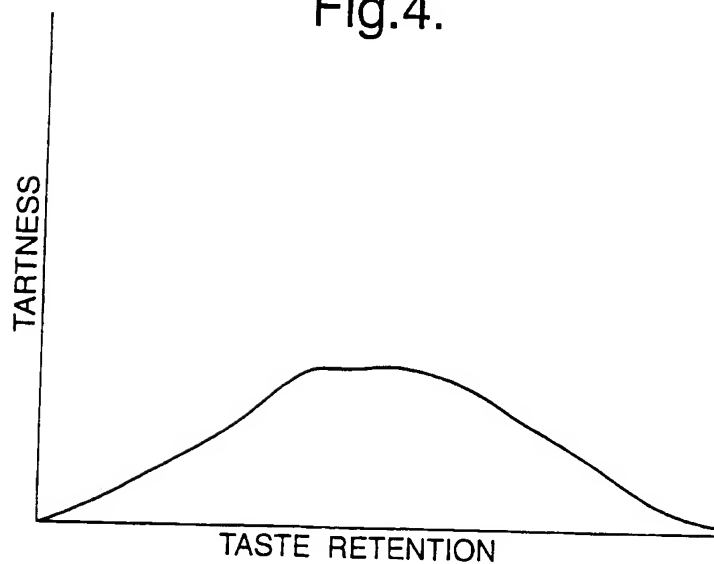


Fig.4.



3/4

Fig.5.

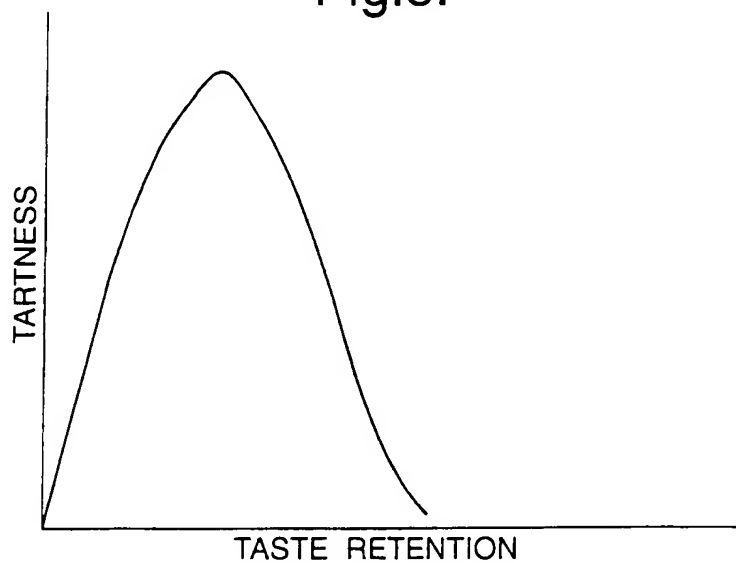


Fig.6.

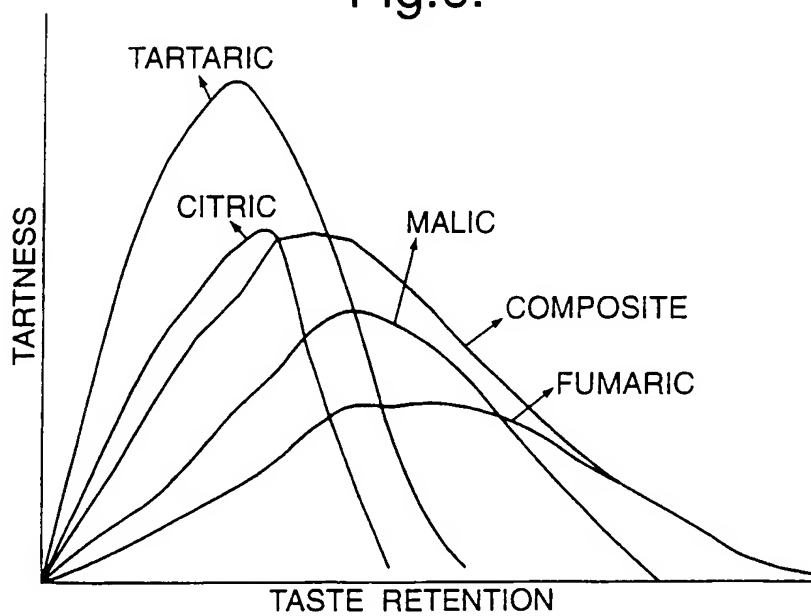


Fig.7.

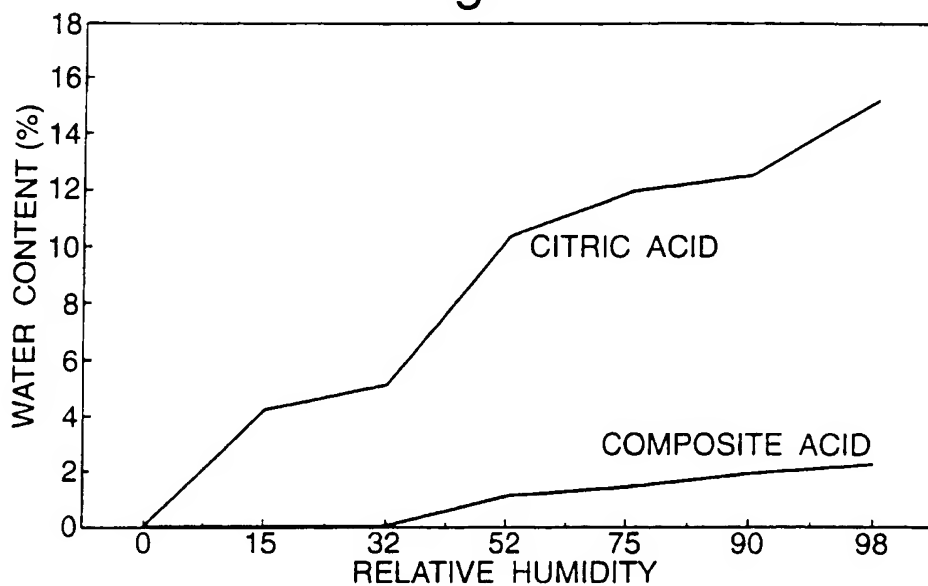
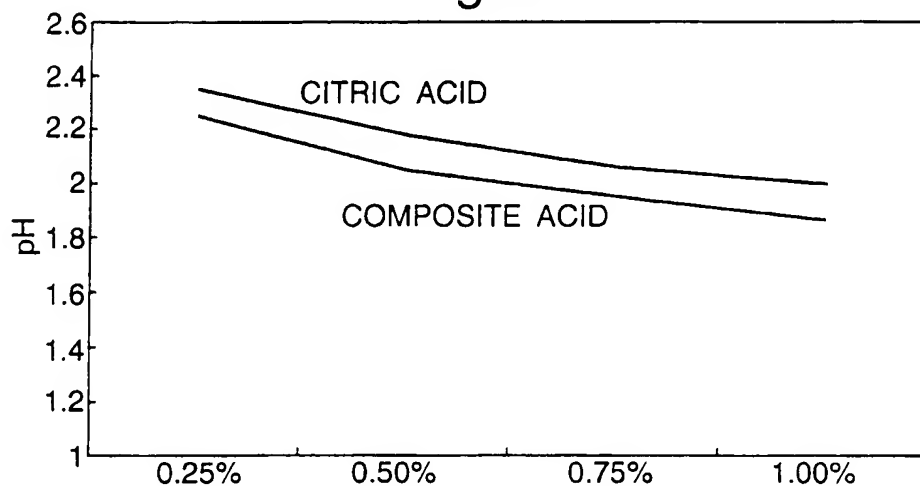


Fig.8.



INTERNATIONAL SEARCH REPORT

Inter. No. National Application No.

PCT/GB 98/01498

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A23L1/22 A23L2/68 A23P1/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	US 3 506 453 A (F.H. MCCARRON) 14 April 1970 see claims ---	1-33
A	US 3 580 726 A (C. DAME) 25 May 1971 see claims; examples ---	1-33
A	GB 1 030 988 A (MONSANTO) 25 May 1966 see page 2, line 13-20; claims ---	1-33
A	US 3 782 974 A (J.F. LONTZ ET AL.) 1 January 1974 see claims -----	1,33

☐ Further documents are listed in the continuation of box C

☒ Patent family members are listed in annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

5 November 1998

Date of mailing of the international search report

11/11/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Van Moer, A

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 98/01498

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